



# Ultrasonic removal of pyridine from wastewater: optimization of the operating conditions

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**Abstract** In this study, a fundamental research had been carried out to explore the removal of pyridine in wastewater by ultrasound radiation. The effects of initial pyridine concentration, radiation time, pH, aeration, and the reaction temperature on the pyridine removal efficiency were investigated. The removal rates of pyridine at 180 min sonication time were found to decrease from 53 to 15 % with increasing the initial concentration from 10 to 100 mg/L. However, the total amount of pyridine degraded after 60 min at 100 mg/L was as much as three times larger than that degraded at 10 mg/L. The optimal pH was found to be 9 which resulted in 25 % pyridine removal after 180 min ultrasound radiation. By observing the change of pH value with the sonication time up to 60 min, it was observed that, pH of the sonicated pyridine aqueous mixture has decreased from 9.2 to 6.2 during the irradiation. The decrease in the pH may be attributed due to the formation of peroxy radicals in the solution and subsequently formation of oxygen free radicals. The simultaneous aeration could improve pyridine removal efficiency of ultrasound irradiation by 24 %. With increasing media temperatures, the removal efficiencies of pyridine increased in the temperature range in this study. In the end, it could be proposed that ultrasound radiation was an effective method for the removal of pyridine from wastewater.

**Keywords** Pyridine · Ultrasound radiation · Wastewater treatment · pH · Degradation

## Introduction

The increasingly acceptable use of chemicals in industries and households has resulted in the increasing generation of organic pollutants in effluents. These pollutants are potential health hazards. Enormous quantities of aromatic compounds as pollutants are being released into the environment by various industries (Jain et al. 2004), because of the broad range of applications of aromatic compounds among the top chemicals used in many industries (Gogate and Pandit 2004). Out of this aromatic heterocyclic compounds such as pyridine and its derivatives are of major concern as environmental pollutants due to their recalcitrant, toxic and teratogenic nature (Stapleton et al. 2006; Karthikeyan et al. 2012).

Pyridine was produced from coal tar and as a by-product of the coal gasification. However, increased demand for pyridine resulted in the development of more economical methods of synthesis from acetaldehyde and ammonia, and more than 20,000 tons per year are manufactured worldwide. Thus, researchers have long sought to develop effective, economically feasible techniques for cleaning the atmosphere of waste such as pyridine (Gupta et al. 2007a, b; Mittal et al. 2008; Saleh and Gupta 2012).

Various physicochemical methods for the wastewater treatment have been investigated (Goel et al. 2004; Mittal et al. 2010; Gupta et al. 2012). These include adsorption (Gupta et al. 2006, 2009, 2011), electrochemical (Gupta et al. 2007c), sorption using waste material (Gupta and Sharma 2003; Gupta et al. 2006, 2010; Mittal et al. 2010), biodegradation (Padoley et al. 2006; Mathur et al. 2008), and ozonation with biodegradation (Ince and Tezcanlı 2001; Agustina et al. 2005). After extensive search, a lack in literature reports on the removal of pyridine from wastewater by ultrasound radiation has been determined.

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Nowadays, ultrasonic irradiation has received considerable interest as an advanced oxidation process because it leads to rapid degradation of chemical contaminants in water (Abbasi and Asl 2008; Yang et al. 2009). Ultrasound can enhance or promote chemical reactions and mass transfer and offer the potential for shorter reaction cycles, cheaper reagents, and less extreme physical conditions (Gong and Hart 1998; Goel et al. 2004). So far, ultrasound has been applied in studies of cleaning, organic synthesis, catalysis, extraction, emulsification, material processing, food processing, and wastewater treatment (Vinodgopal et al. 1998; Naffrechoux et al. 2000; Saleh and Gupta 2012).

In this study, an investigation was focused on the ultrasonic degradation of pyridine in aqueous media. A variety of different operating conditions were examined. The effects of different initial concentration, pH, aeration, and reaction temperatures on the degradation efficiency were investigated.

## Materials and methods

### Materials

Pyridine standard solution was supplied by Fluka with purity better than 98.0 %. It was used to prepare a synthetic wastewater. Aqueous solutions were made using deionized water, which was prepared by an Elga B114 Deionizer using C114 cartridges, (EC 5  $\mu\text{S cm}^{-1}$  5 °C and TDS 3.5 ppm). All other reagents were reagent grad obtained from Fluka and used as received.

### Ultrasonic setup reactor

The degradation experiments were carried out in ultrasonic cleaner bath (Honda electronics PS-60, Capacity 15 L). The bath operates at 360 W and 40 kHz. Erlenmeyer flask was used as reaction vessel. The volume of the solution was 100 mL. The bath temperatures were maintained by proper recirculation of water. Solution temperature was also monitored regularly. The efficiency of a reaction vessel placed in an ultrasonic bath depends strongly on the distance of the bottom of the reaction vessel to the bottom of water bath. The distance was carefully measured through preliminary experiments, so that ultrasonic intensity reached maximum at the bottom of the flask. For ultrasonic frequency 40 kHz this distance value was 1 cm. The reactor was sealed with silicone stopper wrapped with an aluminium foil to ensure the minimum loss due to evaporation of the volatile compounds. The syringe needle was pierced through the septum of the stopper for sampling. In the present study,

ultrasonic device provides indirect sonication, which will inevitably cause energy loss. A limited quantity of energy is transmitted into the reaction vessel. So, it should be kept in mind that the power used is not the real power of ultrasonic energy transmitting into the reaction mixture. All sonochemical experiments were conducted twice in parallel. The averages of the parallel experimental data were calculated and taken into account in analyses of sonochemical kinetics. The error of all parallel experiments was under 5 %.

### Quantitative analysis experiments

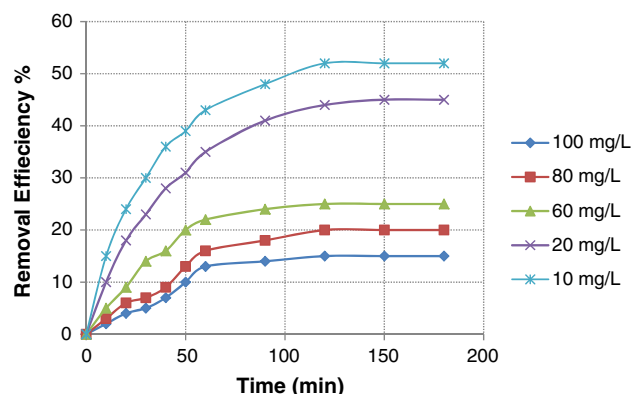
Quantitative analysis of Pyridine concentration was determined by measuring its absorbance using Shimadzu UV–Visible spectrophotometer. Initially, tests were carried out by UV scans from a wavelength of 200–500 nm to determine the absorption maxima of the pyridine molecule.

## Results and discussion

### Study and optimization of the operating conditions

#### *Effect of initial pyridine concentration*

The effect of solute concentration on the degradation of pyridine was investigated at pH 6.7, 40 kHz,  $20 \pm 1$  °C, 360 W and initial concentration 10, 20, 60, 80 and 100 mg/L. It follows from the data obtained in Fig. 1 that degradation of pyridine depends upon sonication time. The removal rates of pyridine at 180 min sonication time were found to decrease from 53 to 15 % with increasing the initial concentration from 10 to 100 mg/L. This suggests that increasing the initial concentration of the solution would decrease the removal rates of pyridine. This is because the increment of initial concentration of the



**Fig. 1** Pyridine removal efficiencies versus time by ultrasound at various initial concentrations

volatiles results in the weakening effect of cavitation reactions (Stapleton et al. 2007). However, the total amount of pyridine degraded after 60 min at 100 mg/L was as much as three times larger than that degraded at 10 mg/L.

The degradation rates can be expressed by the following equation:

$$\ln C_t/C_0 = -Kt \quad (1)$$

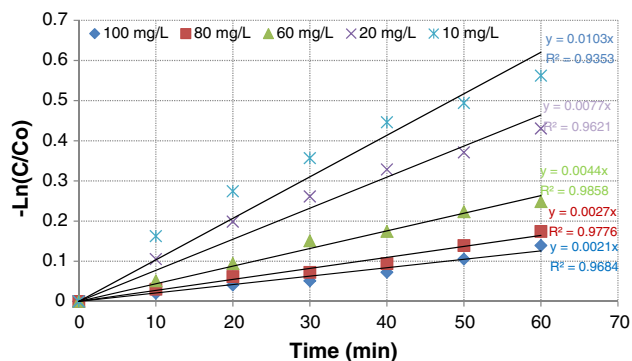
where  $C_0$  and  $C_t$  are the initial and remaining concentrations of pyridine, respectively,  $k$  is the degradation rate constant, and  $t$  is the sonication time.

A typical  $\ln C_t/C_0$  vs.  $t$  Fig. 2 show that, the reaction kinetics for the degradation followed first order rate laws ( $R^2 > 93$ ) at all initial concentrations. In addition to, as it is seen from the Fig. 3, the apparent first order rate constants decreased with an increasing initial concentration of the pyridine, indicating non-elementary nature of the sonochemical reactions. This dependence of reaction rate constants on initial concentration compared well with existing literature (Zechmeister and Magoon 1956; Stapleton et al. 2006, 2007).

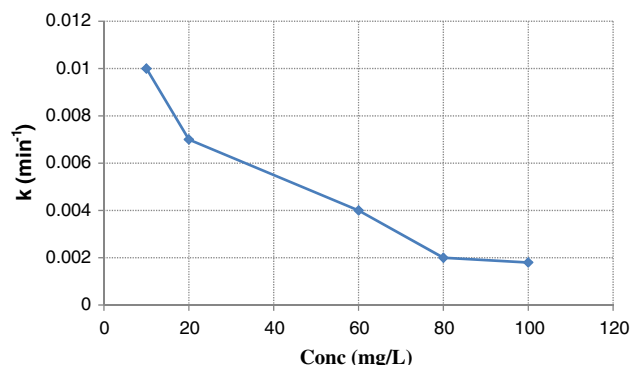
Sonochemical degradation is based on physicochemical processes that produce in situ powerful free radical species,

principally hydroxyl radicals ( $\text{HO}\cdot$ ), using chemical and/or other forms of energy, and have a high efficiency for organic matter oxidation;. It is highly powerful oxidizing agent having an oxidation potential of 2.33 V, which can undergo rapid and non-selective reaction with most organic and many inorganic pollutant. Hydroxyl radicals exhibit faster rates of oxidation reactions comparing to conventional oxidants such as  $\text{H}_2\text{O}_2$  or  $\text{KMnO}_4$ . Once generated, the hydroxyl radicals can attack organic chemicals by radical addition. The reaction conditions vary as the concentration of monocyclic aromatic compounds in aqueous solution changes. In our case, products formed by the degradation of pyridine should affect the reaction rate due to their influence on the cavitation temperature; however, it is difficult to evaluate and establish (De Visscher et al. 1996).

It seems that the major route for degradation of pyridine during ultrasonic irradiation alone without any additives is by pyrolytic reactions in the gas phase, and thus it shows a greater dependence on initial concentration. Ultrasonication not only promotes oxidative degradation of pyridine by hydroxyl radicals, but also provides a possible route for thermal decomposition in the gas phase (Naffrechoux et al. 2000).



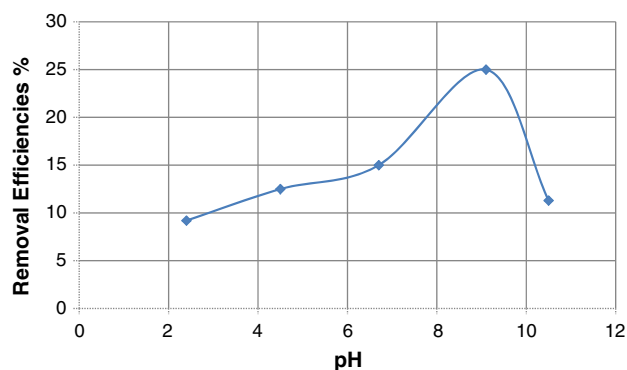
**Fig. 2** The first order kinetic of pyridine degradation (40 kHz,  $20 \pm 1$  °C and 360 W)



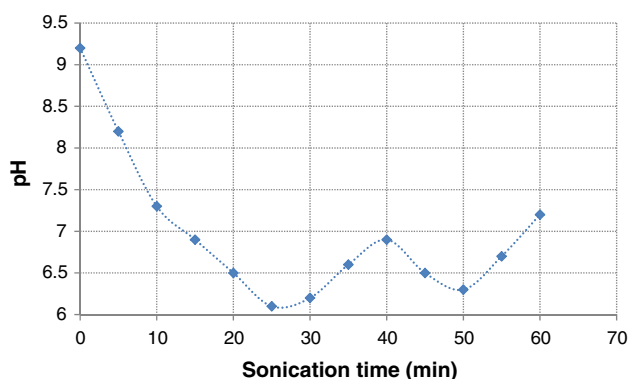
**Fig. 3** Apparent first order rate constants vs. initial concentration of pyridine (40 kHz,  $20 \pm 1$  °C and 360 W)

#### Effect of initial pyridine solution pH

In this part of study, sonication experiments were repeated with pyridine solutions 100 ppm to study the effect of pH on the degradation reaction. The initial pH of the solution was adjusted by adding 1–3 drops of NaOH (0.1 M) and HCl (0.1 M). Figure 4 illustrates the removal of pyridine at different pH initial values. It is seen that pH value greatly affects the removal efficiencies for pyridine. The optimal pH was found to be 9 which resulted in 25 % pyridine removal after 180 min pyridine ultrasound (US) radiation. No significant increase of pyridine removal was observed when solution pH was further increased over 9. However, at acidic medium (pH < 6.5) the removal efficiency decreased considerably. This could be attributed to that, Pyridine is a heterocyclic nitrogenous compound and during its degradation, the N atom in the pyridine ring upon mineralization is released as ammonia which easily observed by its unpleasant odor. However, because pyridine contains an N atom, which is more electronegative than an  $\text{SP}^2$  hybridized C atom, it is suggested that at higher acidity values, the formation of pyridinium salt predominate, which is more stable in solution. In addition to, in alkaline medium, the anionic state of compound favors the ultrasonic absorption and production of more hydroxyl radical from hydroxyl ion ( $\text{OH}^- \rightarrow \text{OH}\cdot$ ), which causes the enhancement in degradation efficiency.



**Fig. 4** Effect of the pH on the removal efficiencies of pyridine by ultrasound (40 kHz,  $20 \pm 1$  °C and 360 W)



**Fig. 5** Variation of pyridine solution pH with sonication time (40 kHz,  $20 \pm 1$  °C and 360 W)

On the other hand, by observing the change of pH value with the sonication time up to 60 min, it was observed that pH of the sonicated pyridine aqueous mixture has decreased from 9.2 to 6.2 during the irradiation (Fig. 5). Initially at zero time, pH of the aqueous mixture is found to be 9.2, which has reduced to 6.2 in the first 25 min of sonication. The decrease in the pH may be attributed due to the formation of peroxy radicals in the solution and subsequently formation of oxygen free radicals and the ( $H^+$ ) ions (Sistla 2005). Many researchers reported the possibility of release of [ $H^+$ ] ions after 10 min of irradiation in water. After 15 min of sonication, pH values obtained are nearing 7.2 (Stapleton et al. 2007; Sistla and Chintalapati 2008).

The effect of pH value could be interpreted that pH value affects the distribution of the existing state of the pyridine and all kinds of organic compounds in the wastewater. It is well known that pH value can affect the physicochemical properties of substances in aqueous solution, and thus it is expected that pH value can affect the ultrasonic decomposition rates of substance s in the solution (Xu et al. 2005). This dependence of degradation on pH compared well with existing literature. Kotronarou et al. (1991) and Jain et al. (2004) investigated the influence of the changes in the initial

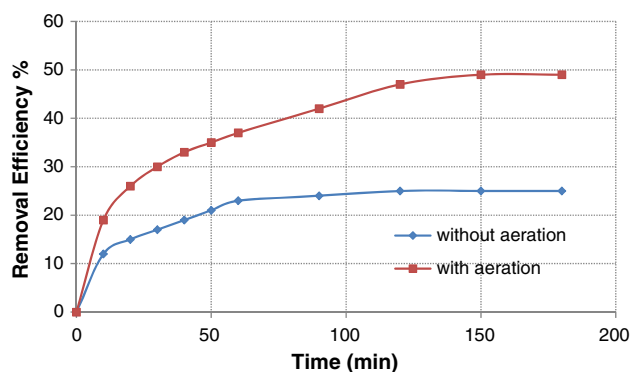
pH of the pnitrophenol (PNP) solutions on the decay of PNP. PNP decayed exponentially with time at all pH values. The pseudo-first order rate constant decreased with the increase of pH (pH 5 to 8), and remained constant up to pH 10. At pH > 10, the pseudo-first order rate constant increased slightly because of the slow thermal reaction between PNP and OH radical/ $H_2O_2$ .

Furthermore, Drijvers et al. (1996) found that the degradation of trichloroethylene is fastest in basic solutions. However, no influence of the pH value of the aqueous solutions on the sonolysis of chlorobenzene was found (Drijvers et al. 1998).

In conclusion, at acidic medium, the formation of salts reduces the vapor pressure of the reactants to such an extent that they are unable to enter the bubbles present and are, hence, unaffected by the ultrasonic waves (Currell et al. 1963).

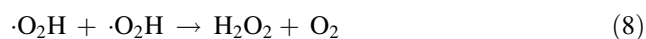
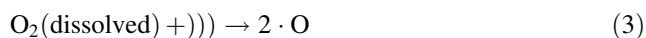
#### *Effect of ultrasound (US) radiation with and without aeration*

The cavitation effect of ultrasound causes the degassing of liquids. Therefore, many researchers deliberately bubble gas through a sonochemical reaction to facilitate uniform cavitation (Kotronarou et al. 1991). To determine the effect of dissolved gas, experiments were carried out in air saturated solutions. For these experiments, the test gas was introduced into the reactor during the sonication experiments  $1.5 \text{ L min}^{-1}$ . Figure 6 illustrates the removal of pyridine with and without aeration for pyridine concentration 100 ppm, pH 9.1. It could be seen that the removal was enhanced by aeration to some extent. Aeration brought a lot of air bubbles into the solution. This might result in turbulence and agitation. Therefore, the mass transfer in the solution was enhanced, which benefited the volatile of molecular decomposition product. So, the simultaneous aeration could improve pyridine removal efficiency of ultrasound irradiation by 24 %. Though the oxidation of organic pollutants by air is minimal, aeration could disturb pyridine strongly and may even break the surface continuity of pyridine, thus resulting in more cavitation nuclei in pyridine and much more efficient mass transfer and so more degradation of organic pollutants. When ultrasound irradiation is combined with aeration, aeration increases the concentration of cavitation bubbles in solution, enhancing the effective utilization of ultrasound energy, accelerating cavitation and pyrolysis. And at the same time, the cavitation bubbles are broken into ‘mini-bubbles’. The total surface area of mini-bubbles is more than 103–104 times higher than that of cavitation bubbles (Xu et al. 2005). So the interfacial area between air and water was increased, and pyridine removal increased.



**Fig. 6** Effect of dissolved gas on the degradation of 100 ppm pyridine solution (40 kHz,  $20 \pm 1$  °C, pH 9.1 and 360 W)

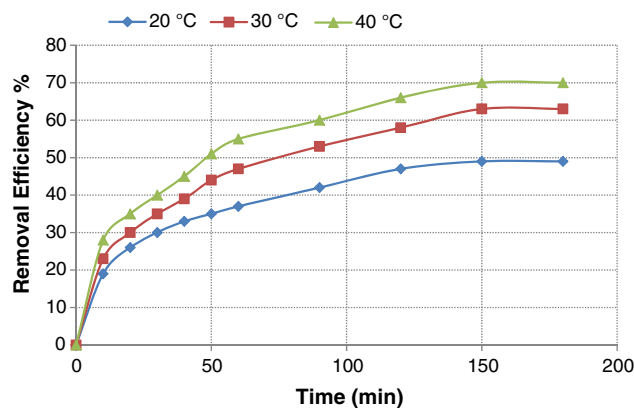
On the other hand, If the solution is saturated with oxygen, additional reactions occur as a consequence of combination of molecular oxygen with hydrogen atoms and the thermal decomposition of oxygen in the gas phase according to Eqs. (2)–(8) (Pang et al. 2011). In these reactions “)))” denotes the US irradiation. These reactions produce higher hydroxyl radical concentration, which cause more degradation of pyridine



Furthermore, many investigators have extensively stated that dissolved gases are essential for the sonochemical reaction. Griffing (2004) noted that the rate of the hydrolysis of  $\text{CCl}_4$  is strongly dependent on the dissolved gas. Weissler et al. (1950) confirmed the effect of dissolved gases ( $\text{O}_2$ ,  $\text{N}_2$ , He,  $\text{CO}_2$ , vacuum) on the iodine yields during the ultrasonic irradiation of KI solutions in the absence of  $\text{CCl}_4$  and with a large excess of  $\text{CCl}_4$ .

#### Effect of sonochemical reaction temperature

The effect of media temperature on pyridine degradation was investigated at an initial pyridine concentration of 100 ppm, pH of 9.1, air flow at  $1.5 \text{ L min}^{-1}$ , and power of 360 W. Three different temperatures were utilized to investigate the influence of operating temperature on the cavitation reactions. The media temperature in solution gradually increases when ultrasound starts to operate, due to the thermal energy from ultrasound irradiation; therefore a water circulating system was needed to keep the operating temperature stable. Actual



**Fig. 7** Effect of media temperature on the degradation of the pyridine (pyridine concentration 100 ppm, air flow  $1.5 \text{ L min}^{-1}$ , pH 9.1, 40 kHz and 360 W)

temperatures measured inside of the reaction container were 20, 30, and 40 °C. Temperature error was manipulated within 2 °C. The results are illustrated in Fig. 7. With increasing media temperatures, the removal efficiencies of pyridine increased in the temperature range in this study. Generally speaking, the chemical reaction rate usually increases as temperature rises, the liquid temperature and applied pressure dramatically affect the sonochemical reaction rates. The bulk temperature and static pressure first affect the vapor pressure, gas solubility, components in cavities, and thermal activation. Thereby, the intensity of collapse and the secondary reaction rate are influenced (Naffrechoux et al. 2000).

On the other hand, the effective maximum temperature generated during the cavitation collapse is inversely proportional to the vapor pressure. A bubble contains not only the gas that is dissolved in the liquid, but also vapor from the liquid itself. The amount of vapor in the bubble depends on the vapor pressure of the liquid, which is strongly dependent on the temperature of the bulk liquid. Combining all of the above, the effect of temperature on sonochemical degradation rate is complicated. Thus, there is no consistent report on the impact of temperature on the degradation of organic compounds in literature. Bhatnagar and Cheung (1994) reported that the degradation of trichloroethylene and carbon tetrachloride remained constant between  $-7$  to  $20$  and  $20$ – $60$  °C, respectively. In contrast, Destailats et al. (2000) indicated that the sonochemical degradation of chlorobenzene and TCE, respectively, increased with increasing temperature. In this study, slightly higher degradation is achieved at higher temperatures.

#### Conclusion

Ultrasonic irradiation has the potential for use in environmental decontamination due to the production of high



concentrations of oxidizing species such as  $\cdot\text{OH}$  and  $\text{H}_2\text{O}_2$ , in the solution and localized transient high temperatures and pressures. It does not require the addition of chemical additives to achieve viable degradation rates. However, by carefully adjusting the operating conditions, the degradation efficiency can be significantly increased. In this study, sonochemical degradation of pyridine under different process parameters was conducted. Effects of different process variables such as initial concentration, pH, aeration, and media temperature were tested. The reaction rate was observed to be a function of the initial concentration of the pyridine. It decreases with the increase in initial concentration. The degradation efficiency of pyridine increases with the increasing of the reaction temperature, the initial pH, and aeration. The research has shown that it is technically feasible to decompose pyridine compound by sonolysis. The advantage of ultrasonic degradation lies in the amount of energy stored in the microbubbles. If properly utilized, it can be a truly useful technology for large scale water treatment. The process is easier to operate and there are practically no hazards associated with it.

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